



REC'D 28 SEP 2004

WIPO

PCT

Patent Office
Canberra

**PRIORITY
DOCUMENT**

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND
SALES hereby certify that annexed is a true copy of the Provisional specification
in connection with Application No. 2003905081 for a patent by RESEARCH
LABORATORIES OF AUSTRALIA PTY LTD as filed on 18 September 2003.



WITNESS my hand this
Twentieth day of September 2004

A handwritten signature in cursive script, reading "J. Billingsley".

JULIE BILLINGSLEY
TEAM LEADER EXAMINATION
SUPPORT AND SALES

BEST AVAILABLE COPY

RESEARCH LABORATORIES OF AUSTRALIA PTY LTD

AUSTRALIA
PATENTS ACT 1990

PROVISIONAL SPECIFICATION FOR THE INVENTION ENTITLED:
" METHOD OF PREPARTION AND PRODUCT PRODUCED THEREBY "

This invention is described in the following statement:

FIELD OF THE INVENTION

This invention relates to a method of preparation of electrostatic marking liquids for use in printers.

5 BACKGROUND OF THE INVENTION

Electrostatography is a term used to describe various non-impact printing processes which involve the creation of a visible image by the attraction of charged imaging particles or marking particles to charge sites present on a substrate. Such charge sites, forming what is usually termed a latent image can be transiently supported on
10 photoconductors or pure dielectrics and may be rendered visible in situ or be transferred to another substrate to be developed in that location. Additionally such charged sites may be the reflection of those structured charges existing within a permanently polarised material as in the case with ferroelectrics or other electrets.

15 Electrostatography encompasses those processes normally known as electrophotography and electrography.

In general, the process of production of marking liquids commences with a resin and a colourant, which can be extruded from a suitable mixing machine or by other
20 techniques known to the art. The colourant can be a dye which is soluble in the resin or a pigment comprising of colourant particles which are not soluble in the resin.

The extrudate is ground coarsely and then milled in a carrier liquid in which neither the resin nor the colourant is soluble, to produce a marking liquid with very fine marking
25 particles distributed in it.

So produced marking liquids have been found to have a rheology which can have non-Newtonian flow with applied shear and hence may not have ideal flow characteristics suitable under all conditions for their ultimate intended use. Particle size distribution of
30 the marking particles of so produced marking liquids has also been found to be variable. Other problems encountered include poor dispersion stability, variable electrical characteristics and generally variable print performance.

It is the object of this invention to provide a process for producing these marking liquids which will overcome these problems by providing an alternative ink or toner preparation method.

5 BRIEF DESCRIPTION OF THE INVENTION

In one form, although this may not necessarily be the only or broadest form the invention is said to reside in a method of preparation of a liquid electrostatographic toner or liquid ink jet ink, the method including the steps of,

- 10 a) preparing an extrudate of a resin and a colourant and coarse grinding the extrudate,
- b) milling the coarse ground particles of colourant and resin with a carrier liquid to produce a liquid marking particle mix,
- c) heating the marking particle mix to a temperature at or above the glass transition temperature of the resin of the marking particle mix,
- 15 d) maintaining the temperature of the heated marking particle mix for a selected period of time,
- e) cooling the resin to room temperature, and
- f) mixing the marking particle mix with high shear.

- 20 The selected period of time may be from several minutes to several days depending upon the type of heating applied and the method of applying that heat.

- Heating can be done by convection, such as in an oven, conduction or radiation, such as microwave radiation. Due to the different energies of these different heating systems
25 the method chosen will alter the heating time and temperature required to achieve the desired results.

- Preferably the step of milling the resin and colourant particles includes milling with additives which can include charge control agents and dispersion agents as is known to
30 the art.

In a further form the invention may be said to reside in a method of preparation of a liquid electrostatographic toner or liquid ink jet ink, the method including the steps of,

- a) heating a marking particle mix including a carrier liquid and a resin which is insoluble in the carrier liquid to a temperature at or above the glass transition temperature of the resin of the marking particle mix,
- b) maintaining the temperature of the heated marking particle mix for a selected period of time,
- c) cooling the marking particle mix to room temperature, and
- d) mixing the marking particle mix with high shear.

The invention also resides in a liquid electrostatographic toner or liquid ink jet ink prepared by any of the methods discussed above.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other objects, features and advantages of the present invention will become more apparent from the following detailed description taken with the accompanying drawings in which:

FIG. 1 is a graph illustrating the rheology of Example 1 prior to treatment.

FIG. 2 is a graph illustrating the rheology of Example 1 after treatment.

FIG. 3 is a graph illustrating the rheology of Example 2 prior to treatment.

FIG. 4 is a graph illustrating the rheology of Example 2 after treatment.

DETAILED DESCRIPTION OF THE INVENTION

In this specification, the invention will be discussed particularly in relation to the use of the invention for the production of liquid toners, including so-called high viscosity high concentration liquid toners, for electrostatic printing applications. It would be understood by those skilled in the art that the methods described herein are also applicable to the production and processing of ink jet inks, including inks suitable for electrostatic type ink jet printers.

In general, a liquid developer, or toner, for electrostatography is prepared by dispersing an inorganic or organic colourant in a carrier liquid. The liquid developer should be

stable, not only in terms of suspension stability, but also of electrical charge. As such numerous additional components can be additionally integrated into the developer to achieve liquid developers that exhibit reproducible high quality images.

5 In such developers, it has been recognised that certain electrical properties of the carrier liquid are mandatory requirements for the effective functioning of a conventional electrostatographic liquid development process. The mandatory requirement is low electrical conductivity but other requirements have also become obvious, such as the need for low toxicity, increased fire safety, low solvent power, low odour etc. For these reasons, isoparaffinic-hydrocarbons such as the Isopar® range manufactured by Exxon Corporation, the Shellsol® range manufactured by Shell Chemical and the Soltrol® range manufactured by Phillips Petroleum have become the industry standards for liquid toner carriers.

15 Other carrier liquids may be used, and these may also comprise a silicone fluid of straight chained configuration, a silicone fluid of cyclic configuration, a silicone fluid of branched configuration, or a combination thereof.

The carrier liquid may also comprise a vegetable oil. Representative examples of vegetable oils include soybean oil, cottonseed oil, safflower oil, sunflower oil, castor oil, linseed oil and olive oil.

The carrier liquid may also comprise a synthetic oil. Representative examples of synthetic oils include fatty esters obtained by the reaction between higher fatty acid and alcohol, and ester compounds obtained by the reaction between higher fatty acid and ethylene glycol or glycerine.

The carrier liquid may also comprise a polybutene, a synthetic hydrocarbon polymer made by the polymerisation of isobutene (also known as "isobutylene"). Another name for polybutene is polyisobutylene.

It would be understood by those skilled in the art that blends of the abovementioned

carrier liquids or other suitable carrier liquids could be used in relation to this invention.

5 Marking particles that are insoluble in the carrier liquid may be selected upon their particular proposed end use. Examples of marking particles include inorganic pigments such as carbon blacks, silica, alumina, titanium dioxide, magnetic iron oxide, or organic pigments such as phthalocyanine blue, alkali and reflex blue, phthalocyanine green, diarylide yellow, arylamide yellow, azo and diazo yellow, azo red, rubine toner, quinacridone red, basic dye complexes, lake red, or fluorescent pigments and dyestuffs
10 such as basic dyes and spirit soluble dyes, or combinations thereof. Other materials, as would be understood by those skilled in the art, could be used as marking particles.

As indicated above, the liquid developer or toner may also include an organic or
15 inorganic insoluble marking particle and such a marking particle may be present in the range of 2 to 50 percent by weight.

The liquid toner may also contain therein additional components including but not limited to resins and binders, specific examples of which can include: ethyl cellulose,
20 linseed-oil-modified alkyd resin, acrylic or methacrylic ester resin, polystyrene, silicone-acryl copolymer, silicone resin, silicone-(meth)acryl copolymer, block polymer or graft polymer, polyolefin copolymer, poly(vinyl chloride) resin, chlorinated polypropylene, polyamide resin, coumarone-indene resin, rosin-modified resin, and alkylphenol-modified xylene resin, synthetic polyesters; polypropylene or modified polypropylene;
25 alkylated poly vinyl pyrrolidones; natural waxes such as montan wax, candelilla wax, sugar cane wax, beeswax, natural resins such as ester gum and hardened rosin; natural-resin-modified cured resins such as natural resin-modified maleic acid resins, natural resin-modified phenol resins, natural resin-modified polyester resins, natural resin-modified pentaerythritol resins and epoxy resins. Other components such as
30 plasticisers can also be incorporated, examples of which are sulfonamides, adipates, sebacates and phthalates.

Additionally to affect or enhance electrostatic charge on such dispersed particles additives known as charge directors or charge control agents may be included. Such materials can be metallic soaps, fatty acids, lecithin, organic phosphorus compounds, succinimides, sulphosuccinates etc.

5

The charge control agent may be present in a range of 0.01 to 5 percent by weight of the toner when used.

10

The liquid developer or toner may also include a dispersant which can be selected, for example, from the Solsperse range of polymeric hyperdispersants including 13940 made by Avecia; amino-silicones including Finish WR1101 made by Wacker Chemicals; polymeric oil additives including Plexol made by Rohm and Haas; polymeric petroleum additives including FOA-2 made by Dupont; multi-functional pigment dispersing agents including Disperse Axd 1 made by Elementis DCP Inc.

15

20

The dispersing agent may be incorporated into the liquid composition by techniques commonly employed in the manufacture of liquid compositions such as: ball-jar milling, attritor milling, bead milling etc. Pre-mixing techniques involving blending the dispersion agent into the carrier liquid before the addition of marking particles and before the milling stage can also be used to incorporate the dispersion additive into the liquid developer formulation.

25

The dispersant may be present in a range of 0.1 to 20 percent by weight of the toner when used.

30

Examples of suitable liquid toner formulations include those disclosed in commonly assigned U.S. Patent 5,591,557, U.S. Patent 5,612,162, U.S. Patent 6,174,640 and U.S. patent 6,287,741, the disclosures of which are totally incorporated herein by reference.

The Applicant has surprisingly found that liquid toners manufactured as in the abovementioned disclosures will exhibit an enhanced performance on all levels if the so produced liquid toner is post treated by heating the liquid toner to a temperature at or

above the glass transition temperature, or the so-called first softening point, of the resin of the marking particle mix, maintaining the temperature of the heated marking particle mix for a selected period of time, cooling the resin to room temperature, and mixing the liquid toner with high shear.

5

The Applicant has also found that liquid toner performance can be even further enhanced by matching the liquid toner resin system to the dispersing agent and carrier liquid. The heating process can then be optimised to the thermal characteristics of the resin system, and therefore produce a more effective post treatment result.

10

Although the exact process by which this method of preparation improves the desired characteristics of a liquid toner is not fully understood, the following explanation will be given but the Applicant is not bound to this explanation.

15 It is believed that during the heating stage when the resin is heated to at or just above its glass transition temperature, the structure of the resin particles begins to relax as internal stresses are released as the resin begins to transit through its glassy state, and surface tension effects occur such that the morphology of the toner particles assumes a smoother shape.

20

The action of a suitable dispersing agent in the liquid toner prior to the treatment is useful at this point because it ensures that individual particles of resin and colourant remain separated from one another so as they do not agglomerate or stick together. The dispersing agent achieves this through its strong affinity for the toner particle causing it
25 to form a protective layer around the particle and therefore providing an adequate barrier between neighbouring resin and colourant particles through steric hindrance forces and enabling the particle surface to relax and flow and thus become smoother.

30

It has been noted that after the heating and cooling stage, a weakly formed gel structure can often form which is believed to be due to possible weak attraction or interaction of dispersion agent species residing on the surface of one particle with that of another

particle. This interaction is easily broken up by moderate shearing forces used during the high speed mixing stage of the process.

It has also been noted that liquid toners produced by the process of the present invention exhibit a more Newtonian like flow behaviour with very little or no yield viscosity, as can be see in FIG. 2 and FIG. 4, even with high solid content formulations of up to 50% by weight solids but preferably in the range of 20 - 40% weight solids. There is a significantly reduced high shear toner viscosity. This means that when the toners for instance are used in printing machines, the characteristic viscosity of the toner is less dependant on the speed of rollers and the like and hence has a more even and predictable action during use.

Smoothing of the toner particle surface results in a reduced surface area causing improved flow properties which can be related to the measured viscosity change with change in shear.

The applicant has also found improved toner stability. Depending on the type of dispersion agent and resin system used, there can be an enhanced interaction during the heating stage of the process between the dispersion agent and the particle and/or the reaction of amino functional groups of specific dispersion agent materials with epoxide groups of specific resin system to key the dispersion agents strongly to the surface of the toner particle resulting in improved toner dispersion stability. Increased stability may also be achieved as a result of a reduced toner particle surface area such that the ratio of dispersion agent available per unit surface area of toner particle has substantially increased.

The applicant has also noted improved toner electrical properties. Due to smoothing of the toner particle surface, reduced high shear viscosity and Newtonian like flow behaviour being achieved, the charge to mass ratio of the toner particles increases apparently due to higher absorption of charging and dispersing species on the toner surface along with increased toner mobility to give improved electrical properties to the liquid.

The electrical characteristics can be measured with a liquid toner characterization cell, of which apparatus details are disclosed in commonly assigned U.S. Patent Application Publication 2001/0035352A1, the disclosure of which is totally incorporated herein by reference.

5

The liquid toners prepared according to this present invention also exhibit improved print performance. Toners and inks prepared according to the present invention show substantially increased optical density, decreased background staining, higher image resolution and improved toner management and handling characteristics. These toner
10 handling and management characteristics include improved ability for recycling and replenishment of toners and the general flow of toner within a printer.

This then generally describes the invention but to assist with understanding, reference will now be made to the accompanying non-limiting examples which shows
15 embodiments of the invention.

The various examples were tested using an electrostatic printer of the type described in patent specification WO95/08792.

20 EXAMPLE 1

An extrudate was prepared with the following composition:

Extrudate 1

	Epikote 1001	61.5g
	Antaron V220	18.5g
25	Irgalite Blue LGLD	20g

Epikote 1001 is an epoxy resin made by Shell Chemicals, Australia. Irgalite Blue LGLD is a CI Pigment Blue 15:3 made by Ciba-Geigy, Basel Switzerland. Antaron V220 is an alkylated polyvinylpyrrolidone made by GAF/ISP Chemicals, New Jersey U.S.A.

30

A marking liquid of the following composition was prepared using the Extrudate 1:

Extrudate 1	125g
-------------	------

Finish WR1101		5g
DC 200 Fluid	10 cSt	370g

5 Finish WR1101 is a dimethyl polysiloxane having amino-alkyl functional groups, made by Wacker Chemicals, Munich Germany. DC 200 10cSt Fluid is a silicone fluid made by Dow Corning, U.S.A.

EXAMPLE 2

A marking liquid of the following composition was prepared using the Extrudate 1:

10	Extrudate 1		125g
	Finish WR1101		5g
	DC 200 Fluid	50 cSt	370g

DC 200 50cSt Fluid is a silicone fluid made by Dow Corning, U.S.A.

15 The marking liquids of the above two examples were prepared by adding the constituents into a ceramic ball jar containing spherical ceramic grinding media and milling for 4 days to prepare a resinous toner.

It should be understood that the quantities of raw materials in the Example can be varied dependent on the liquid developer characteristics required and the mode of
20 operation of the electrostatic printer.

The marking liquids were then divided into two batches. A first batch of each was further treated according to the present invention as follows:

The so produced toner was placed in a suitable container and subjected to heating in an
25 oven. The liquid toner was heated in the oven at a temperature of 60°C and maintained at that temperature for a period of 48 hours without any form of agitation.

The thermal properties of the Extrudate 1 resin system are as follows:

Extrudate 1	Glass Transition (T _g)	Melt (T _m)
Temperature	50.9 ^o C	87.2 ^o C

The above results were measured using a Seiko Thermo Mechanical Analyser (TMA),

5 Type TMA120C.

Heating of the liquid toner to 60^oC, being above the glass transition temperature of the resin of the marking particle mix, is such that the structure of the resin particles begin to relax as internal stresses are released and surface tension effects occur such that the morphology of the toner particles assumes a smoother shape. After the 48 hour heating period, the liquid toner was taken out of the oven and allowed to cool to room temperature. Once the liquid toner is at or near room temperature the liquid toner is then mixed with high shear. This was achieved with a T.K. Homo Mixer, type M, manufactured by Tokushu Kika Kogyo Co. Ltd. The toner is subjected to high shear mixing for 2 minutes at 10,000 rpm.

The selected heating period may be from several minutes to several days depending upon the type of heating applied and the method of applying that heat. Heating can be done by convection, such as in an oven, conduction or electromagnetic radiation, such as microwave radiation. Due to the different energies of these different heating systems the method chosen will determine the conditions required to achieve the desired results. In some instances however, such as in a convection oven, it has been found that it is important that the liquid toner is evenly heated to the required temperature, and that no or minimal agitation takes place during the heating stage. The heating temperature can be dependent on the resin mixture used in the liquid toner formulation and the Applicant has found that it should be at or just above the glass transition temperature of the resin material.

High shear mixing can be achieved with any commercially available high shear mixers. The applicant has found that the degree of mixing required is dependent on the solid

content of the formulation, that is, the higher the solids content of the liquid developer the higher the energy input requirement.

The batches both before and after the treatment of the invention were then examined for various physical properties and for print quality by producing images with the abovementioned electrostatic printer.

Physical Properties

Particle size.

- Characteristics measured were $D[4,3]$ which indicates the equivalent spherical volume diameter mean and this value is biased toward larger particles since volume is a function of the cube of the particle radius; and $D[v,0.5]$ which indicates the volume 50% value of the distribution. This differs from $D[4,3]$ if the volume distribution is skewed.

- Results were as follows:

Example 1	Before Treatment	After Treatment
$D[4,3]$	1.27 μm	1.10 μm
$D[v,0.5]$	1.06 μm .	0.93 μm

Example 2	Before Treatment	After Treatment
$D[4,3]$	1.95 μm	1.79 μm
$D[v,0.5]$	1.51 μm .	1.44 μm

These results show a significant drop in particle size and a narrower distribution. Particle size distribution was measured using a Malvern Mastersizer.

Viscosity

- Viscosities were measured using a HAAKE RheoStress RS100. All measurements were taken at 20°C. FIG. 1 of the attached drawings shows Example 1 before treatment, and

FIG. 2 shows Example 1 after treatment.

FIG. 1 illustrates that prior to the treatment, the toner was non-Newtonian and exhibited shear thinning, with a high shear viscosity at 3000 s^{-1} of 92.1 mPa.s . FIG. 2 illustrates the same toner after the treatment. It now exhibits a significant reduction in non-Newtonian flow behaviour or yield viscosity, with a high shear viscosity at 3000 s^{-1} of 53.2 mPa.s .

FIG. 3 of the attached drawings shows Example 2 before treatment, and FIG. 4 shows Example 2 after treatment.

FIG. 3 illustrates that prior to the treatment, the toner was non-Newtonian and exhibited shear thinning, with a high shear viscosity of 200 mPa.s . FIG. 4 illustrates the same toner after the treatment. It now exhibits a significant reduction in non-Newtonian flow behaviour or yield viscosity, with a high shear viscosity of 137 mPa.s .

Dispersion Stability

Examples 1 and 2 were examined before and after treatment for dispersion stability by assessment of agglomeration and sedimentation over a specified time period.

Sedimentation was assessed by samples being placed in a volumetrically graduated sedimentation flask and the % of sedimentation analysed after a defined time period; the amount of decrease in the meniscus represents the separated (sedimented) solids compared to the original meniscus volume. Agglomeration was assessed by samples being placed in beaker for a specified period of time and then assessed by stirring samples gently. The agglomeration level can be determined by the samples resistance to stirring.

It was found that over a 6 month storage period, dispersion stability had been greatly improved with the treated samples, as can be seen in the following results:

Example 1	Sedimentation	Agglomeration
No treatment	>31%	moderate
Treatment	<4%	none

5

Example 2	Sedimentation	Agglomeration
No treatment	>22%	moderate
Treatment	<2%	none

Electrical characteristics

10 It is well known that the electrical properties of liquid toners used in electostatography significantly influence the quality of a printed image; the most important electrical characteristics of conventional liquid toners are known to be conductivity, electrophoretic mobility and zeta-potential. Testing of these as well as other electrical characteristics of Example 1 and Example 2 both prior to and post treatment show improved characteristics which are reflected in the improved toner performance in the

15 abovementioned electrostatic printer.

Results were as follows:

Example 1	Before Treatment	After Treatment
Mobility	$7.0 \times 10^{-11} \text{ m}^2\text{N/s}$	$2.4 \times 10^{-10} \text{ m}^2\text{N/s}$
Conductivity	313.8 pS/cm	389.5 pS/cm
Zeta Potential	42 mV	145 mV

Example 2	Before Treatment	After Treatment
Mobility	$5.4 \times 10^{-11} \text{ m}^2\text{N/s}$	$1.0 \times 10^{-10} \text{ m}^2\text{N/s}$
Conductivity	0.23 pS/cm	22.7 pS/cm
Zeta Potential	162 mV	300 mV

Print Testing

- 5 The samples were tested for image optical density and background staining using the abovementioned printing device.

The results were as follows:

Example 1	Before Treatment	After Treatment
Optical Density	1.25 +/- 0.18 ODU	1.85 +/- 0.19 ODU
Background Staining	0.07 +/- 0.03 ODU	0.00 +/- 0.00 ODU

10

Example 2	Before Treatment	After Treatment
Optical Density	1.32 +/- 0.16 ODU	2.02 +/- 0.17 ODU
Background Staining	0.04 +/- 0.03 ODU	0.00 +/- 0.00 ODU

- 15 As can be see from the above results, treatment resulted in significantly increased optical density in print sample image areas, as well as decreased or no staining or background scatter in non-image areas.

20 There has been hereto described a novel method of preparation of electrostatic marking liquids exhibiting substantially improved imaging performance, including physical as well as electrical stability. The instant invention also describes a method by which prior art electrostatic marking liquids with a compatible resin system can be substantially enhanced in imaging performance, including physical as well as electrical stability.

It can be appreciated that changes to any of the above embodiments can be made without departing from the scope of the present invention and that other variations can be made by those skilled in the art without departing from the invention as defined in
5 the appended claims.

The claims defining the invention are as follows:

1. A method of preparation of a liquid electrostatographic toner or liquid ink jet
10 ink, the method including the steps of,
 - a) preparing an extrudate of a resin and a colourant and coarse grinding the extrudate,
 - b) milling the coarse ground particles of colourant and resin with a carrier liquid to produce a liquid marking particle mix,
 - 15 c) heating the marking particle mix to a temperature at or above the glass transition temperature of the resin of the marking particle mix,
 - d) maintaining the temperature of the heated marking particle mix for a selected period of time,
 - e) cooling the marking particle mix to room temperature, and
 - 20 f) mixing the marking particle mix with high shear.
2. A method as in Claim 1 wherein the selected period of time is from several minutes to several days depending upon the type of heating applied and the method of applying that heat.
25
3. A method as in Claim 1 wherein the heating is provided by convection, such as in an oven, conduction or radiation, such as microwave radiation.
4. A method as in Claim 1 wherein the step of milling the resin and colourant
30 particles includes milling with additives which can include charge control agents and dispersion agents.

5. A method as in Claim 1 wherein the resin is selected from ethyl cellulose, linseed-oil-modified alkyd resin, acrylic or methacrylic ester resin, polystyrene, silicone-acryl copolymer, silicone resin, silicone-(meth)acryl copolymer, block polymer or graft polymer, polyolefin copolymer, poly(vinyl chloride) resin, chlorinated polypropylene, polyamide resin, coumarone-indene resin, rosin-modified resin, and alkylphenol-modified xylene resin, synthetic polyesters; polypropylene or modified polypropylene; alkylated poly vinyl pyrrolidones; natural waxes such as montan wax, candelilla wax, sugar cane wax, beeswax; natural resins such as ester gum and hardened rosin; natural resin-modified cured resins such as natural resin-modified maleic acid resins, natural resin-modified phenol resins, natural resin-modified polyester resins, natural resin-modified pentaerythritol resins and epoxy resins.

6. A method as in Claim 1 wherein the colourant is selected from inorganic pigments such as carbon blacks, silica, alumina, titanium dioxide, magnetic iron oxide, or organic pigments such as phthalocyanine blue, alkali and reflex blue, phthalocyanine green, diarylide yellow, arylamide yellow, azo and diazo yellow, azo red, rubine toner, quinacridone red, basic dye complexes, lake red, or fluorescent pigments and dyestuffs such as basic dyes and spirit soluble dyes, or combinations thereof and present in the range of 2 to 50 percent by weight.

7. A method as in Claim 1 wherein the carrier liquid is selected from isoparaffinic-hydrocarbons, silicone fluids of straight chained configuration, silicone fluids of cyclic configuration, silicone fluid of branched configuration, vegetable oils, synthetic oils or a polybutenes or blends thereof.

8. A method as in Claim 4 wherein the charge control agent is selected from metallic soaps, fatty acids, lecithin, organic phosphorus compounds, succinimides and sulphosuccinates and may be present in the range of 0.01 to 5 percent by weight of the toner when used.

9. A method as in Claim 4 wherein the dispersion agent is selected from polymeric hyperdispersants, amino-silicones, polymeric petroleum additives, polymeric oil additives and multi-functional pigment dispersing agents.

5 10. A method of preparation of a liquid electrostatographic toner or liquid ink jet ink, the method including the steps of,

a) heating a marking particle mix including a carrier liquid and a resin which is insoluble in the carrier liquid to a temperature at or above the glass transition temperature of the resin of the marking particle mix,

10 b) maintaining the temperature of the heated marking particle mix for a selected period of time;

c) cooling the marking particle mix to room temperature, and

d) mixing the marking particle mix with high shear.

15 11. A method as in Claim 10 wherein the selected period of time is from several minutes to several days depending upon the type of heating applied and the method of applying that heat.

12. A method as in Claim 10 wherein the heating is provided by convection, such as
20 in an oven, conduction or radiation, such as microwave radiation.

13. A liquid electrostatographic toner or liquid ink jet ink prepared by the method of any one of claims 1 to 9.

25 14. A liquid electrostatographic toner or liquid ink jet ink prepared by the method of any one of claims 10 to 12.

Dated this 18th day of September, 2003.

30 By its Patent Attorneys
MADDERNS



FIG 1.

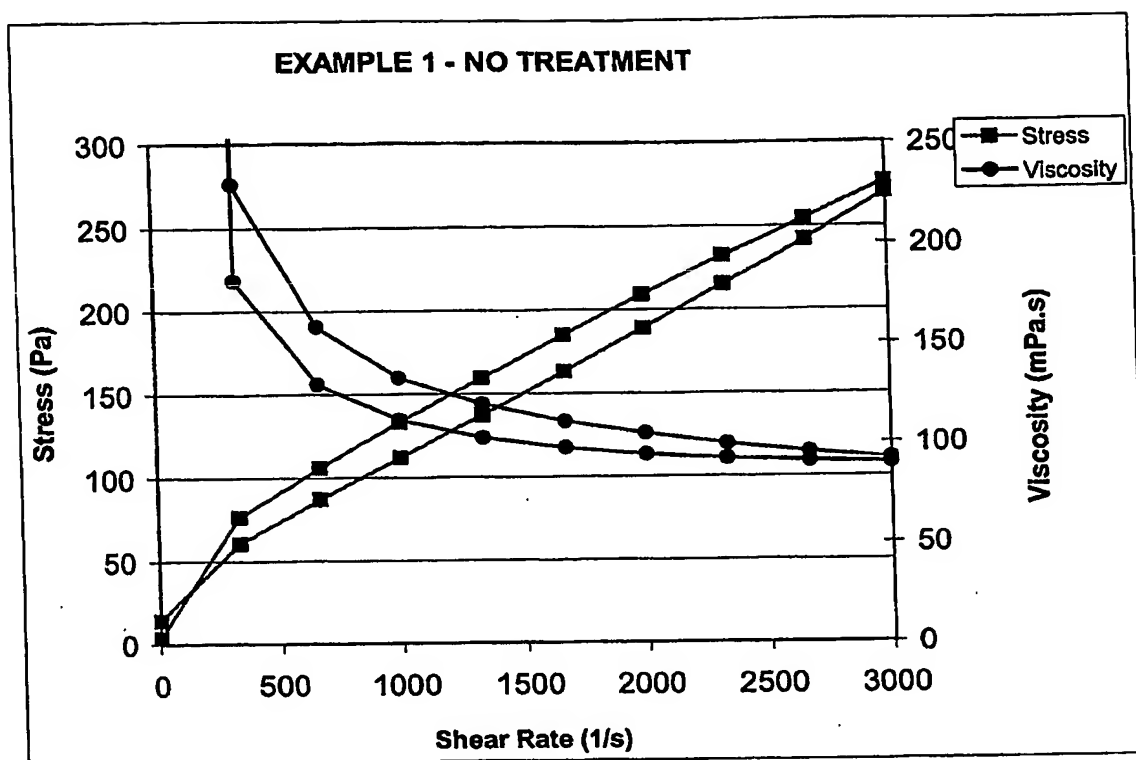


FIG. 2

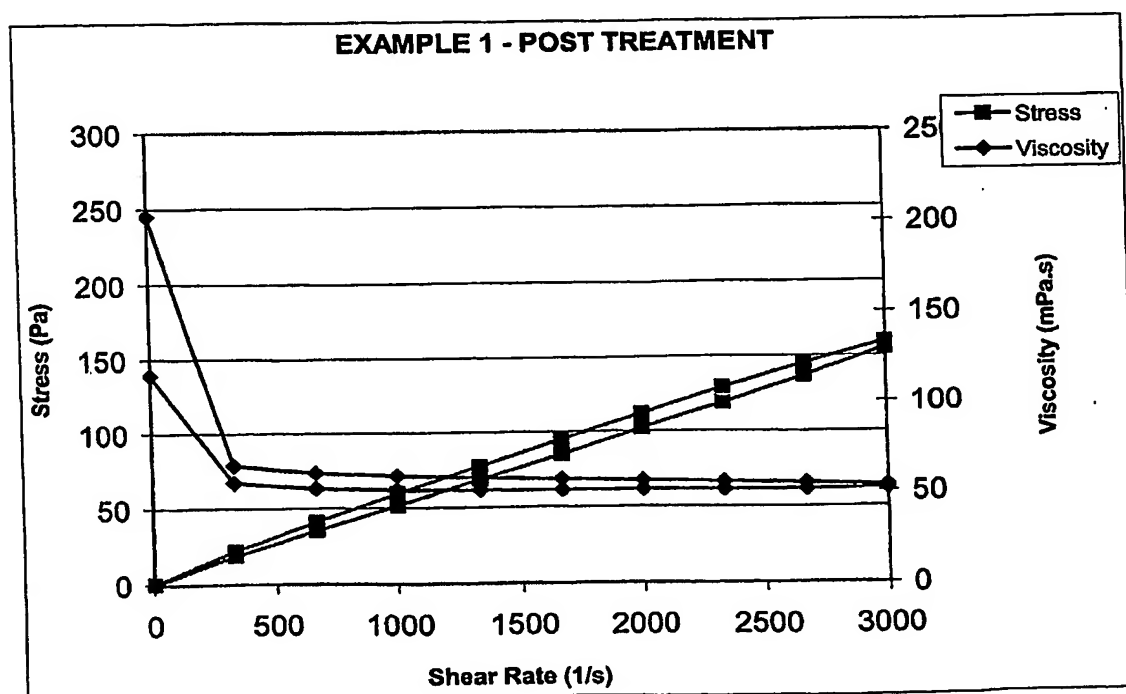


FIG. 3

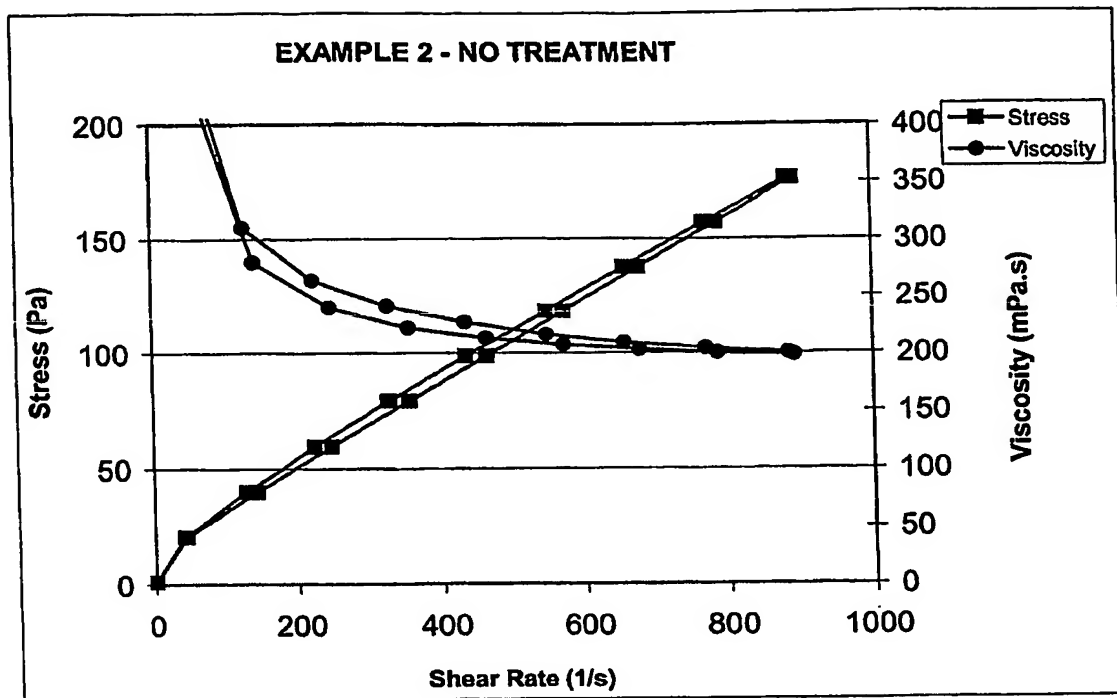
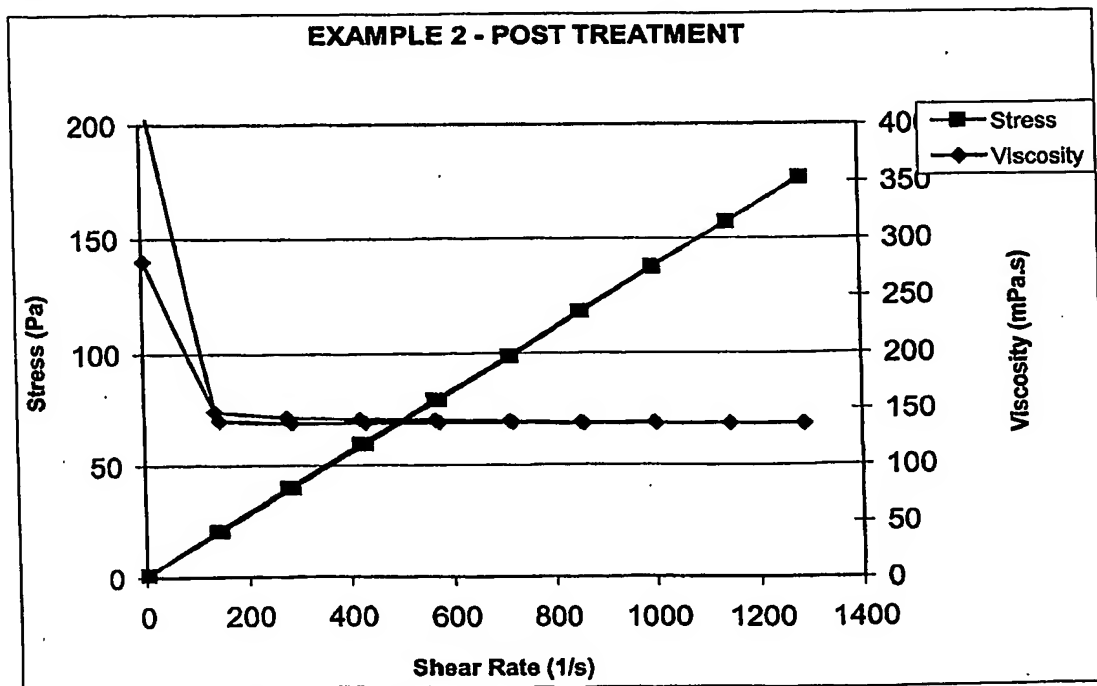


FIG. 4



**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.